DYEING BEHAVIOR PREDICTION OF COTTON FABRICS IN SUPERCRITICAL CO_2

by

Juan ZHANG, Lai-Jiu ZHENG*, Yao-Hua SU, Miao LIU, Jun YAN, and Xiao-Qing XIONG

Liaoning Provincial Key Laboratory of Ecological Textile,
Dalian Polytechnic University, Dalian, China

Original scientific paper
https://doi.org/10.2298/TSCI160805062Z

This paper aims at establishing a model predicting the dyeing behavior of cotton fabrics in supercritical CO_2 by using the multiple linear regression, where dyeing temperature, dyeing pressure, dyeing period, and dye concentration are selected as independent variables. The theoretical prediction shows good accuracy for color strength value and breaking strength of the dyed cotton fabrics.

Key words: *cotton, supercritical CO_2, dyeing, multiple linear regression*

Introduction

Replacing organic solvent or water by supercritical CO_2 ($scCO_2$) is extremely advantageous to energy and resource preserving and environmental protection, for example, short dyeing time, recycling of dyes and CO_2, and zero waste water emission in textile dyeing procedures [1-3]. To date, coloration of synthetic fibers in $scCO_2$, for instance polyethylene terephthalate, polylatide, polyamide 6 and 66, and meta-aramid, has achieved satisfactory results, and even some attempts for commercialization in practice are available [4-6].

Moreover, considerable researches on the dyeing of natural fibers in $scCO_2$ have also been done in modified cotton fibers and reactive disperse dyes for wool, silk, and cotton fibers/fabrics [7, 8]. However, dyeing cotton fibers/fabrics is difficult in $scCO_2$ and the coloration of cotton is affected by several complex factors. In this work, dyeing temperature, pressure, time, and dye concentration were chosen as the independent variables, and color strength (*K/S* values) and breaking strength were chosen as the dependent variable in the linear regression analysis. The suitable multiple linear regression models to predict the dyeing behavior of cotton fabrics in $scCO_2$ were established.

Experimental

Materials and dyeing procedure

Scoured cotton fabrics were supplied from Liaoning Chaoyi Industry & Trade Group, Liaoning, China, in this study, and the fabric samples with a weight of 115.2 g/m^2 were used in the beam dyeing procedure. Reactive golden yellow K-2RA (≥ 98%) was supplied by Zhejiang Shaoxing Fine Chemical Industry, Zhejiang, China. The purity of CO_2 was 99.9%.

* Corresponding author, e-mail: fztrxw@dlpu.edu.cn

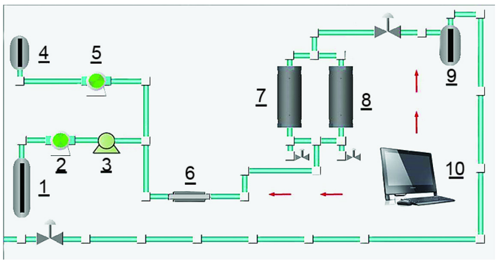

Figure 1. Schematic diagram of $scCO_2$ dyeing apparatus; *1 – gas cylinder, 2 – refrigerator, 3 – high-pressure pump, 4 – cosolvent tank, 5 – cosolvent pump, 6 – heat exchanger, 7 – dye vessel, 8 – dyeing vessel, 9 – separator, 10 – PC*

All the experiments were performed on a batch-type supercritical dyeing apparatus, as shown in fig. 1 [2]. Dyeing of cotton fabrics in $scCO_2$ was carried out from 30-120 minutes at dyeing temperatures, pressures, and dye concentrations ranging from 30-120 °C, 12-30 MPa, and 1-5.5%, respectively.

Multiple linear regression analysis

Multiple linear regression is a common statistical tool for the investigation of relationships between dependent and independent variables. In the multiple linear regression, eq. (1) was employed to express the dependent variable as a function of the independent variables [9]:

$$y = b_1X_1 + b_2X_2 + \ldots + b_nX_n + C \tag{1}$$

where y is the dependent variable, $X_1, X_2,\ldots, X_n$ – the independent variables; $b_1, b_2,\ldots, b_n$ – the regression coefficients, and C is a constant at which the regression line crosses y-axis.

Colorimetric measurement

The *K/S* values of the dyed cotton fabrics were measured by employing a Color-Eye 7000A spectrophotometer (X-rite, Grand Rapids, Mich., USA). The Kubelka-Munk eq. (2) was used to determine the *K/S* values at the wavelength of maximum absorption for each reactive golden yellow K-2RA [10]. The reflectance, R, was obtained at 20 nm intervals in the range of 300-700 nm:

$$K/S = \frac{(1-R_{\min})^2}{2R_{\min}} \tag{2}$$

where K is the absorbance coefficient of the dyed cotton fabrics, S – the scattering coefficient of the dyed cotton fabrics, and $R_{\min}$ – the minimum value of the reflectance curve.

Breaking strength measurement

The breaking strength measurement was carried out on a universal material testing machine (gauge length, 100 mm, test speed, 100 mm per minute, preliminary tension, 2 N, TH-8102S, TopHung, Mudu Town, China). The strength value (σ_b per MPa) was calculated by the eq. (3) [2]:

$$\sigma_b = \frac{4F_p}{\pi D^2} \tag{3}$$

where F_p [kg] is the maximal tensile fracture force, D [cm] – the mean diameter of cotton fibers. Each experiment is measured ten times, and each data of dyed cotton fabrics was the average of ten specimens.

Results and discussion

K/S values model

Data analysis

Dyeing temperature x_1 [°C], pressure x_2 [MPa], time x_3 [minutes] and dye concentration x_4 [%] were used as the independent variables to estimate the *K/S* values y. It proved that there was a nearly linear relationship between the *K/S* values and the dyeing conditions of cotton. Before establishing the prediction model, the *K/S* values of the dyed cotton fabrics had been statistically analyzed to examine the data distribution. The model summary and ANOVA are given in tabs. 1 and 2, respectively.

Table 1. Model summary

Model	R	R^2	Adjusted R^2	Std. error of the estimate
1	0.984	0.968	0.943	0.52233

Table 2. The ANOVA

Model	Sum of squares	D_f	Mean square	F	Sig.
Regression	41.706	4	10.427	38.216	0.01
Residual	1.364	5	0.273		
Total	43.070	9			

As listed in tab. 1, a $R^2 = 0.968$, adjusted $R^2 = 0.943$ and standard error of estimate is 0.52233 presented that the predictive ability of the model was very high. Moreover, the linear regression model was significant, which was confirmed by $F = 38.216$ ($p = 0.01$) in tab. 2.

Model of the multiple linear regression

A high positive correlation was found between *K/S* values and dyeing temperature, pressure, time, and dye concentration. It can be seen from tab. 3 that dye concentration x_4 ($b_4 = 2.782$, $p = 0.002$) and dyeing temperature x_1 ($b_1 = –0.044$, $p = 0.033$) had significant impact on the *K/S* values y. Furthermore, a correlation between *K/S* values y, pressure x_2 ($b_2 = –0.239$, $p = 0.089$), and time x_3 ($b_3 = 1.287$, $p = 0.255$) suggested the possibility of multiple linear regression analysis.

Table 3. Coefficients

Model	Unstandardized coefficients		Standardized coefficients	T	Sig.
	B	Std. error	Beta		
Constant	1.980	1.145		1.730	0.144
x_1	–0.044	0.015	–0.606	–2.920	0.033
x_2	–0.239	0.114	–0.663	–2.108	0.089
x_3	0.018	0.014	0.245	1.287	0.255
x_4	2.782	0.463	1.925	6.013	0.002

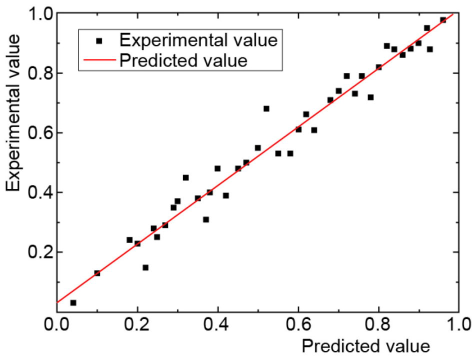

Figure 2. Cross-correlation of experimental and predicted values of the *K/S* values

Based on the previous observations, the multiple linear regression analysis was performed to predict the *K/S* values of the dyed cotton fabrics. The multiple linear regression equation for the prediction of *K/S* values was defined by eq. (4), which displayed a good correlation between *K/S* values and dyeing temperature, pressure, time, and dye concentration:

$$Y = -0.044\,x_1 - 0.239\,x_2 + 0.018\,x_3 + 2.782\,x_4 + 1.980 \qquad (4)$$

Figure 2 showed the scatter plot of the predicted values *vs.* the experimental values, and a good agreement between experimental and predicted results was achieved. The correlation coefficient between the actual and the predicted values was high for the multiple linear regression model. Therefore, it is evident that the predictive ability of the multiple linear regression model is significant.

Breaking strength model

Data analysis

Dyeing temperature [°C], dyeing pressure [MPa], dyeing time [minutes], and dye concentration [%] were used as the independent variables to estimate the breaking strength of the dyed cotton fabrics y [N]. The multiple linear regression equation for the prediction of breaking strength was given:

$$y = -0.893x_1 - 5.248x_2 + 0.594x_3 + 64.356x_4 + 261.185 \qquad (5)$$

The experimental data was subject to regression analysis, and computer output was displayed in tabs. 4-6. The coefficient of multiple linear regression R^2 ($R^2 = 0.963$) and adjusted R^2 ($R^2 = 0.933$) showed that the predictive ability of the model was very high. In addition, it is clear from the results in tab. 5 that the model for breaking strength was significant, which was demonstrated by $F = 32.248$ ($p = 0.001$).

Table 4. Model summary

Model	R	R^2	Adjusted R^2	Std. error of the estimate
1	0.981	0.963	0.933	15.42623

Table 5. The ANOVA

Model	Sum of squares	D_f	Mean square	F	Sig.
Regression	30695.757	4	7673.939	32.248	0.01
Residual	1189.843	5	237.969		
Total	31885.600	9			

Table 6. Coefficients

Model	Unstandardized coefficients		Standardized coefficients	T	Sig.
	B	Std. error	Beta		
Constant	261.185	33.805		7.726	0.001
x_1	–0.893	0.443	–0.454	–2.016	0.100
x_2	–5.248	3.354	–0.534	–1.565	0.178
x_3	0.594	0.407	0.302	1.461	0.204
x_4	64.356	13.666	1.637	4.709	0.005

Model of the multiple linear regression

As listed in tab. 6, dye concentration x_4 ($b4 = 64.356$, $p = 0.005$), dyeing temperature x_1 ($b1 = –0.893$, $p = 0.100$), dyeing pressure x_2 ($b2 = –5.248$, $p = 0.089$), and dyeing time x_3 ($b3 = 1.287$, $p = 0.204$) had important influence on the breaking strength. The cross-correlation between the experimental values and predicted values was illustrated by the multiple linear regression model, as shown in fig. 3.

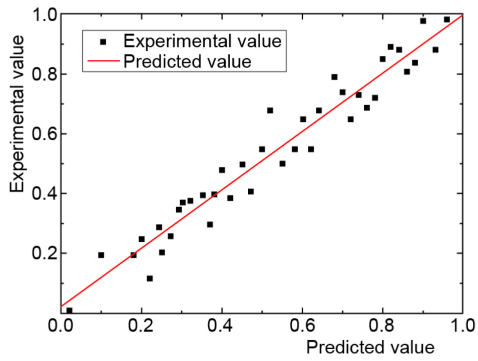

Figure 3. Cross-correlation of experimental and predicted values of breaking strength

Conclusions

The feasibility of dyeing behavior prediction for cotton fabrics using multiple linear regression has been presented in this study. The multiple linear regression models for *K/S* values and breaking strength of the dyed cotton were established in $scCO_2$. The estimation tests indicated that the relationships between *K/S* values and dyeing temperature, pressure, time, and dye concentration were almost linear. On the other hand, breaking strength was highly correlated with dyeing temperature, pressure, time, and dye concentration. Moreover, the regression coefficient R^2 values of the predicted models were very high, and had a significant effect on the dyeing behavior of cotton fabrics in $scCO_2$. There were excellent agreements between the predicted values and the experimental values, proving the validity of the multiple linear regression models.

Acknowledgment

This research was supported by the Program of High-end Foreign Experts Working in the Educational and Cultural Sector (No. GDW20162100068), Group Project of Liaoning Provincial Department of Education (No. 2016J003), and Science Fund from Dalian City (No. 2014A11GX030).

References

[1] Zheng, L., *et al.,* Water in Supercritical Carbon Dioxide Dyeing, *Thermal Science*, *19* (2015), 4, pp. 1301-1304
[2] Zheng, H., Zheng, L, Dyeing of Meta-Aramid Fibers with Disperse Dyes in Supercritical Carbon Dioxide, *Fibers and Polymers*, *15* (2014), 8, pp. 1627-1634

[3] Zheng, H., *et al.,* An Investigation for the Performance of Meta-Aramid Fiber Blends Treated in Supercritical Carbon Dioxide Fluid, *Fibers and Polymers*, *16* (2015), 5, pp. 1134-1141
[4] Zheng, H., *et al.*, An Industrial Scale Multiple Supercritical Carbon Dioxide Apparatus and Its Eco-Friendly Dyeing Production, *Journal of CO_2 Utilization, 16* (2016), Dec., pp. 272-281
[5] Kraan, M., *et al.,* Dyeing of Natural and Synthetic Textiles in Supercritical Carbon Dioxide with Disperse Reactive Dyes, *The Journal of Supercritical Fluids*, *40* (2007), 3, pp. 470-476
[6] Zheng, H., *et al.,* Effect of Treatment Pressure on Structures and Properties of PMIA Fiber in Supercritical Carbon Dioxide Fluid, *Journal of Applied Polymer Science*, *132* (2015), 14, pp. 41756-41762
[7] Zheng, L., *et al.,* Effect of Pressure of Supercritical Carbon Dioxide on Morphology of Wool Fibers During Dyeing Process, *Thermal Science*, *19* (2015), 4, pp. 1297-1300
[8] Zhang, J., *et al.,* Green Dyeing of Cotton Fabrics by Supercritical Carbon Dioxide, *Thermal Science*, *19* (2015), 4, pp. 1283-1286
[9] Montgomery, D., *et al, Introduction to Linear Regression Analysis*, Wiley-Interscience, New Jersey, USA, 2006
[10] Shen, J., *et al.*, On the Kubelka-Munk Absorption Coefficient, *Dyes and Pigments*, *127* (2016), Apr., pp. 187-188

Paper submitted: August 5, 2016
Paper revised: August 26, 2016
Paper accepted: September 4, 2016